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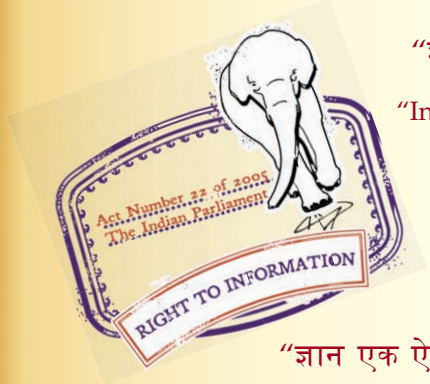
IS 7686 (1975): 3 (N,N-diethyl) Aminophenol [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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IS : 7686 - 1975

Indian Standard

SPECIFICATION FOR 3 (N,N-DIETHYL) AMINOPHENOL

UDC 667·21 : 547·564·4'212

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

July 1975

AMENDMENT NO. 2 NOVEMBER 2003
TO
IS 7686 : 1975 SPECIFICATION FOR 3 (N, N-DIETHYL) AMINOPHENOL

[*Page* 3, *Foreword, Structural formula*] — Insert '(CAS No. 91-68-9)' below structural formula.

[*Page* 4, *Table* 1, *Sl No.* (i) *and* (ii), *col* 4] — Insert 'A-4 'as an alternate method.

(*Page* 7, *clause* **A-3.3**) — Insert the following text after **A 3.3**:

A-4 THIN LAYER CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF IMPURITIES

A-4.1 General

Impurities are determined by thin layer chromatography. Reference may be made to 'IS 5299 : 2001 Methods of sampling and tests for dye intermediates' for details of TLC test method to be followed. However, necessary details of test conditions are given here for guidance only.

1.	Product name	:	N,N-Diethyl-m-aminophenol
2.	Sample solution (on 100% basis)	:	2% Solution acetone + water (8:2)
3.	Application/volume for spotting	:	10 µl for sample and 2 µl and 4 µl for impurities
4.	Standard	:	Reference standard
5.	Test substance for impurities	:	1) m-Amino phenol
			2) Resorcinol (0.05% Solution in Aceton
6.	Plate type	:	Silica gel G
7.	Eluent	:	Toluene : Ethanol 98 : 2 (Saturated — Double run)
8.	Elution time	:	45 min
9.	Temperature	:	25 ± 5°C

10. Detection spray	:	Iodine vapour	
11. Evaluation	:	Semi quantitative	
12. Approx Rf value — Main band	:	N,N-Diethyl-m-aminophenol	: Rf 0.7
—Impurities		m-Aminophenol	: Rf 0.4
		Resorcinol	: Rf 0.3'

(PCD 11)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 APRIL 1990
TO
IS 7686 : 1975 SPECIFICATION FOR 3 (N, N-DIETHYL) AMINOPHENOL

[*Page* 4, *Table* 1, *Sl No.* (ii) , *col* 3] — Substitute ' 98.0 ' *for* ' 97.0 '.

(PCD 11)

Reprography Unit, BIS, New Delhi

Indian Standard

SPECIFICATION FOR 3 (N,N-DIETHYL) AMINOPHENOL

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(*Continued on page 2*)

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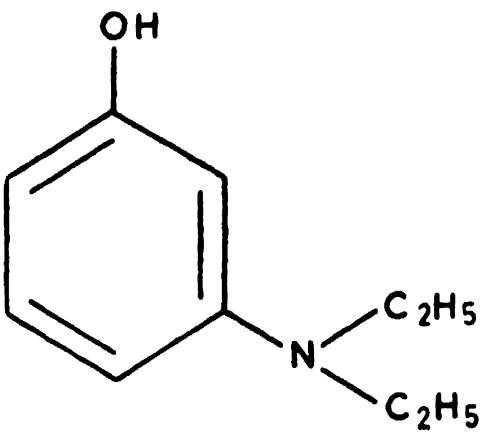
Indian Standard

SPECIFICATION FOR 3 (N,N-DIETHYL) AMINOPHENOL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 18 June 1975, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 3 (N, N-Diethyl) aminophenol ($C_{10}H_{15}ON$) is an intermediate used in the manufacture of dyestuffs. It has the following structural formula:



3 (N, N-DIETHYL) AMINOPHENOL
(Molecular Mass 165·2)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 3 (N, N-diethyl) aminophenol.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white to light brown, moist crystalline solid.

*Rules for rounding off numerical values (*revised*).

2.2 The material shall be soluble in alcohol, ether, sodium hydroxide and hydrochloric acid.

2.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 3(N,N-DIETHYL) AMINOPHENOL

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Appendix A
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C (on dry basis), *Min*	69·5*	A-2
ii)	Assay, percent by mass (on dry basis), *Min*	97·0	A-3

*3(N,N-diethyl) aminophenol has a storage life of about three months from the date of manufacture. On prolonged storage, the material becomes sticky and the crystallizing point is considerably reduced.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Gross, net and tare mass.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests — Tests for the determination of crystallizing point and assay shall be conducted on each of the individual samples.

4.3 Criteria for Conformity — The lot shall be declared as conforming to the requirements of this standard, if the test results as obtained in **4.2** satisfy the corresponding requirements given in Table 1.

5. TEST METHODS

5.1 Tests shall be conducted according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR 3 (N,N-DIETHYL) AMINOPHENOL

A-1. PREPARED SAMPLE

A-1.1 Dry the material in a vacuum oven at 45°C to constant mass and transfer immediately into a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

A-2. DETERMINATION OF CRYSTALLIZING POINT

A-2.1 Determine the crystallizing point of the *prepared sample* (*see* **A-1.1**) as prescribed in **7.1.2** of IS : 5299-1969*.

---

*Methods of sampling and tests for dye intermediates.

†Specification for water, distilled quality (*revised*).

A-3. ASSAY

A-3.0 Outline of the Method — The purity of the material is determined by coupling it with *p*-nitrobenzene diazonium chloride.

A-3.1 Reagents

A-3.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-3.1.2 *p-Nitrobenzene Diazonium Chloride Solution* — 0·1 N. Dissolve 34·5 g of *p*-nitroaniline in 100 ml of concentrated hydrochloric acid and 100 ml of water by heating and dilute to one litre with warm water. Add 200 ml of 0·25 N *p*-nitroaniline solution to a 500-ml volumetric flask cooled to 5°C. Add 50 ml of 1 N sodium nitrite solution rapidly which has been cooled to 5°C. Dilute the resultant solution to 500 ml with water which has been cooled to 5°C. It should give a positive test for nitrous acid when tested with a starch-iodide paper and is ready for use after standing for one or two minutes. Store the solution in an ice-bath in the dark. The solution should be practically colourless and not yellow and it should not be more than slightly turbid. Do not use the solution after standing for more than 5 hours. Standardize the solution freshly before use.

A-3.1.3 *Tetrazodianisidine Solution* — Dissolve 2 g of dianisidine hydrochloride in 7 ml of hydrochloric acid. Heat, if necessary. Cool to 0°C and titrate with 1 N sodium nitrite solution to just completion of reaction. Make up the solution to 100 ml in a volumetric flask. Store this solution in an amber-coloured bottle in a cool place.

A-3.1.4 *H-Acid* (*1-Amino-8-Hydroxy-Naphthalene-3,6-Disulphonic Acid*) *Indicator Solution* — Dissolve 0·5 g of H-acid in 100 ml of 1 percent sodium carbonate solution.

A-3.2 Procedure — Weigh accurately about 5 g of the *prepared sample* (*see* **A-1.1**) into a 500-ml beaker. Add about 200 ml of water and just enough of hydrochloric acid to dissolve the sample. Transfer this solution to a 500-ml volumetric flask quantitatively and dilute up to the mark with water. Take 50 ml of this solution into a 1-l beaker. Add about 45 g of sodium acetate (hydrated) crystals and cool the mixture externally to 10°C. Titrate against *p*-nitrobenzene diazonium chloride solution taken in a jacketted-burette through which ice-cold water is circulated. As the titration progresses, place a drop of the reaction mixture on Whatman No. 1 filter paper (or equivalent) and touch the runout with a drop of tetra-azodianisidine solution. If there is any amount of uncoupled 3 (N, N-diethyl) aminophenol in the reaction mixture, there will be a colour development at the junction of the two solutions. This colour fades

progressively and at a stage it disappears completely. Now touch the runout with the H-acid solution. A pink colour which persists for 5 minutes marks the end point.

A-3.3 Calculation

$$\text{Assay, percent by mass} = \frac{V \times N \times 165}{M}$$

where

V = volume in ml of *p*-nitrobenzene diazonium chloride solution required for the sample,

N = normality of the diazonium chloride solution, and

M = mass in g of the material taken for the test.

INDIAN STANDARDS

ON

AMINOPHENOLS AND AMINOAZOBENZENES

IS :

2630-1975 Nitrobenzene (*first revision*)
2741-1973 β-Naphthol (*first revision*)
2744-1964 α-Naphthylamine
3242-1965 β-Oxy naphthoic acid (BON ACID)
5299-1969 Methods of sampling and tests for dye intermediates
7686-1975 3 (N, N-diethyl) aminophenol

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Printed at Caslon Press, New Delhi, India